

WHAT IS CLAIMED IS

1. A method of preparing and utilizing a catalyst for nano-fiber synthesis, comprising the following steps:
  - a. heating a metal oxide to an initial temperature of  
5 between 400 and 500°C in 10-20% hydrogen at a heating rate of 1-10°C/min to affect its reduction and holding for around 10-60 minutes;
  - b. increasing the temperature to between 550-700°C; and
  - 10 c. passing a mixture of CO/H<sub>2</sub> over the catalyst to produce the nano-carbon fibers.
2. The method in claim 1, wherein the metal oxide comprises iron oxide.
3. The method in claim 1, wherein the metal oxide  
15 comprises a mixture of iron and copper oxides.
4. The method in claim 3, wherein the mixture of iron and copper oxides contains a 99:1 to 50:50 weight ratio of Fe to Cu.
5. The method in claim 1, wherein the metal oxides are  
20 selected from a group consisting of oxides of iron, copper, nickel, molybdenum and combinations thereof.
6. The method in claim 1, wherein the heating time in step (a) is less than 60 minutes.
7. The method in claim 1, wherein steps a and b are  
25 performed in less than two hours time.
8. The method in claim 1, wherein the mixture of CO/H<sub>2</sub> is provided at 1:4 to 4:1 by volume.

9. The method in claim 1, wherein the mixture of CO/H<sub>2</sub> is provided at 1:4 by volume.

30 10. The method in claim 1, wherein the carbon production rate equals or exceeds 2.5 Carbon/g catalyst/hr.

11. The method in claim 1, wherein the method comprises a continuous method for producing catalyst and carbon nano-fibers by reducing the pre-reduction time of the catalyst.

35 12. The method in claim 1, wherein the hydrogen is balanced by an inert gas.

13. A method of producing and utilizing a catalyst for nano-fiber synthesis, comprising the following steps:

40 a. heating a metal oxide catalyst to an initial temperature of between 400 and 500°C in 10% hydrogen at a heating rate of 5°C/min to affect its reduction and holding for less than 60 minutes;

b. increasing the temperature to at least 550°C;

45 c. passing a mixture of CO/H<sub>2</sub> over the catalyst to produce nano-carbon fibers.

14. The method in claim 11, wherein the mixture of CO/H<sub>2</sub> is provided at 1:4 by volume.

50 15. The process in claim 11, wherein carbonaceous feedstock flow to produce nano-fibers begins within one hour from when the metal oxide catalyst is brought to its initial temperature of between 400 and 500°C.

16. A method of producing and utilizing a catalyst for nano-fiber synthesis, comprising the following steps:

55 a. heating a metal oxide catalyst to an initial temperature of between 400 and 500°C in 10-20% hydrogen at

a heating rate of 5°C/min to affect its reduction and holding for around 10-60 minutes;

b. increasing the temperature to at least 550°C but no higher than 700°C;

60 c. passing a mixture of CO/H<sub>2</sub> over the catalyst to produce nano-carbon fibers.

17. The method in claim 16, wherein the method comprises a continuous method of producing the catalyst for nano-fiber synthesis.

65 18. A method of preparing a catalyst for nano-fiber synthesis, comprising the following steps:

a. heating a metal oxide to an initial temperature of between 400 and 500°C in 10-20% hydrogen at a heating rate of 1-10°C/min to affect its reduction and holding for around 70 10-60 minutes; and

b. increasing the temperature of the catalyst to between 550-700°C for use as a catalyst in producing nano-fiber synthesis.

75 19. A method of producing a catalyst for nano-fiber synthesis, comprising the following steps:

a. heating a metal oxide catalyst to an initial temperature of between 400 and 500°C in 10% hydrogen at a heating rate of 5°C/min to affect its reduction and holding for less than 60 minutes; and

80 b. increasing the temperature of the catalyst to at least 550°C for use in producing nano-carbon fibers.

20. A method of producing a catalyst for nano-fiber synthesis, comprising the following steps:

a. heating a metal oxide catalyst to an initial 85 temperature of between 400 and 500°C in 10-20% hydrogen at a heating rate of 5°C/min to affect its reduction and

holding for around 10-60 minutes; and

b. increasing the temperature of the catalyst to at least 550°C but no higher than 700°C so that the catalyst  
90 can be used to produce nano-carbon fibers.

21. The method in claims 18 or 19 or 20, wherein a mixture of CO/H<sub>2</sub> is passed over the catalyst to produce nano-carbon fibers.